IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	HILGREN ET AL.	Examiner:	J. PAK
Serial No.:	09/614,631	Group Art Unit:	1653
Filed:	JULY 12, 2000	Docket No.:	163.1382US01
Title:	METHOD AND COMPOSITION FOR INHIBITION OF MICROBIAL GROWTH IN AQUEOUS FOOD TRANSPORT AND PROCESS STREAMS		

DECLARATION UNDER 37 CFR § 1.131

Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

I, John D. Hilgren, declare and state the following:

1. I am an inventor of the subject matter of the patent application identified above and an employee of Ecolab, Inc., the assignee of the patent application identified above.

2. I understand that the Examiner has cited Hei (US 6,024,986) as prior art in prosecution of the application identified above. I understand that the Hei patent was filed on May 24, 2999.

3. I further understand that the filing date of the present patent application Serial No. 09/614,631 is July 12, 2000.

4. I state that before the date of the Hei patent, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above. We made this invention in the United States.

5. The claims of the patent application identified above relate to compositions including:

about 35 to about 45 weight-% acetic acid, about 5 to about 15 weight-% octanoic acid, about 3 to about 8 weight-% hydrogen peroxide, about 8 to about

16 weight-% peroxyacetic acid, about 1 to about 5 weight-% peroxyoctanoic acid, and about 0.1 to about 2 weight-% chelating agent;

about 40 weight-% acetic acid, about 10 weight-% octanoic acid, about 5 weight-% hydrogen peroxide, about 12 weight-% peroxyacetic acid, about 3 weight-% peroxyoctanoic acid, and about 0.6 weight-% chelating agent;

about 10 to about 150 ppm acetic acid, about 5 to about 40 ppm octanoic acid, about 4 to about 20 ppm hydrogen peroxide, about 5 to about 50 ppm peroxyacetic acid, about 2 to about 25 ppm peroxyoctanoic acid, and about 0.2 to about 2.5 ppm chelating agent;

about 133 ppm acetic acid, about 33 ppm octanoic acid, about 17 ppm hydrogen peroxide, about 40 ppm peroxyacetic acid, about 10 ppm peroxyoctanoic acid, and about 2 ppm chelating agent;

about 50 to about 60 weight-% acetic acid, about 10 to about 20 weight-% octanoic acid, about 5 to about 15 weight-% hydrogen peroxide, and about 0.3 to about 1 weight-% chelating agent; or

about 54 weight-% acetic acid, about 14 weight-% octanoic acid, about 10 weight-% hydrogen peroxide, and about 0.6 weight-% chelating agent;

Each claimed composition also has at least about 1 part by weight of peroxyoctanoic acid for each about 5 parts of peroxyacetic acid.

The present patent application includes at page 15 several tables describing embodiments of the claimed compositions. Two of the tables describe concentrate and use compositions including:

Chemical	Wt-%	ppm
Acetic Acid	40	133
Hydrogen Peroxide	5	17
HEDP	0.6	2
Octanoic Acid	10	33
Peroxyacetic Acid	12	40
Peroxyoctanoic Acid	3	10

One of the tables describes raw materials that can be used to make the concentrate composition described above. These raw materials include:

Raw Material	Weight %
Glacial Acetic Acid	54
Hydrogen Peroxide, 35%	30
HEDP, 60%	1
Octanoic Acid, 95%	15

6. During the research and development leading to the claimed compositions, and before May 24, 1999, my coinventors and I made several compositions meeting the limitations of the claims of the present application and including the compositions described in the tables in paragraph 5 above.

7. Accompanying Exhibit A reports studies of compositions that meet the limitations of the present claims and including the compositions described in the Tables in paragraph 5 above. In Exhibit A, the initial, concentrate, and use compositions described in the tables in paragraph 5 above are referred to as "Falcon 15 O". The composition called "Falcon 15 AE" also meets the limitations of the present claims. The contents of these formulas can be found at page 9-10 of Exhibit A. Page 9 of Exhibit A describes the stability of these formulas. The pages of the report attached as Exhibit A were prepared before May 24, 1999.

8. The evidence presented in Exhibit A indicates that, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above. Moreover, Exhibit A demonstrates the concentrations of the compositions called Falcon 15 O and Falcon 15 AE, as invented prior to May 24, 1999, fit within the claims of the present invention. Pages 1 and 9 of exhibit A also demonstrates the compositions of Falcon 15 invented prior to May 24, 1999, were physically stable.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

John D. Hilgren

EXHIBIT A

DATED MAY 25, 2006

APPLICATION NO. 09/614,631

TITLE PS-13 FALCON 15 PROJECT NO. 34

W-299 BOOK NO. 9980

Work continued from Page

OBJECTIVE: Develop a concentrated version of Falcon KX-6049. Or develop a sanitizer concentrate that maximizes actives (POAA, POOA, H_2O_2) while minimizing cost.

Discussion w/ [redacted]: [redacted] has developed a "Falcon 15" formula that is physically stable: [redacted] Formula A)

	AM	*Equilibri
Glacial Acetic Acid	59.0%	50.87
H_2O_2 (35%)	25.0	3.50
Dequest 2010 (60% Active)	1.0	0.60
Octanoic Acid	15.0	12.28
~~Acet~~ POAA	0.0	10.30
POOA	0.0	3.02
Water	0.0	19.67

*The Equilibrium concentrations were based on the "SOLVER" computer program. We chose equilibrium constants based on @ 3 samples of Falcon 15 ~~produced~~ analyzed by analytical. $K_1 = 1.7$ & $K_2 = 2.35$

FALCON 15 EQUILIBRIUM CONSTANTS DERIVATION

RICHTER/REINHARDT FORMULA

	% Raw Mat'l
Acetic Acid	59.00
Hydrogen Peroxide	8.75
Dequest 2010 (60%)	0.60
Octanoic Acid	15.00
POAA	
POOA	
Water	16.65
	100.00

[redacted] Sp. Grav. 1.0443

Concentration in Molarity

	9807-48	9889-36	9889-46
AAi	10.27	10.27	10.27
H2O2i	2.69	2.69	2.69
OAi	1.09	1.09	1.09
H20i	9.66	9.66	9.66
AAeq	8.98	8.04	7.78
H2O2eq	1.02	0.99	1.01
OAeq	0.87	0.78	0.76
POAAeq	1.34	1.43	1.39
POOAeq	0.30	0.20	0.19
H2Oeq	10.56	14.88	16.16
K1 =	1.54	2.69	2.85
K2 =	3.54	3.95	4.05
K3 (from eq)	2.30	1.47	1.42

ANALYTICAL RESULTS

**After Manipulation of SOLVER Program we decided: K1 = 1.7 & K2 = 2.35 — using these Keq's.

K1=2.77 K2=4.00 calculated based on Analytical

2 Week Equilibrium Concentrations (% By Weight)

9807-48	9889-36	9889-46	SOLVER	SOLVER
51.57	46.19	44.70	50.87	49.54
3.33	3.21	3.29	3.50	2.62
0.60	0.60	0.60	0.60	0.6
12.00	10.80	10.50	12.28	11.76
9.73	10.41	10.10	10.30	11.98
4.57	3.14	2.95	3.02	3.6
18.20	25.65	27.86	19.67	20.13
100.00	100.00	100.00	100.24	100.23

$AA + H_2O_2 \xrightarrow{K1} POAA + H_2O$

$OA + H_2O_2 \xrightarrow{K2} POOA + H_2O$

$POAA + OA \xrightarrow{K3} POOA + AA$

K1 ave of last two: 2.77
K2 ave of last two: 4.00
K3 ave of last two: 1.44

SIGNATURE [redacted] 10.01.16

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According to [redacted], original goal was to maximize octanoic acid content in raw material but they determined a solubility limit near 15%. Physical stability tests are performed at 40°F and 100°F.

It appears from Micro testing that the lower the H_2O_2 the better. We will focus on ① Minimizing cost ② maintain Physical Stability ③ Maintain Chemical Stability ④ Maximize PA/PA+H_2O_2 ratios ⑤ Understand other factors involved that increases efficacy. ⑥ Make Non-Detonable Solutions.

I. Physical Stability TESTS

A) CONCENTRATE
① Room Temp.
② 40°F
③ 100°F - 1 MONTH?
I. Qualitative - Turbidity
II. Quantitative - G.C

B) Use Solution - May use Analytical G.C. To determine if octanoic separates to the top.
① Room Temp
② 40°F
③ 100°F

II. Chemical Stability TESTS
- CONCENTRATE ONLY
① Room Temp.
② 100°F

Method of Analysis
① QATM 202
③ HPLC.

III. EFFICACY
⑥ - At use concentrations
- 4 exposure times / Temps. / Etc.

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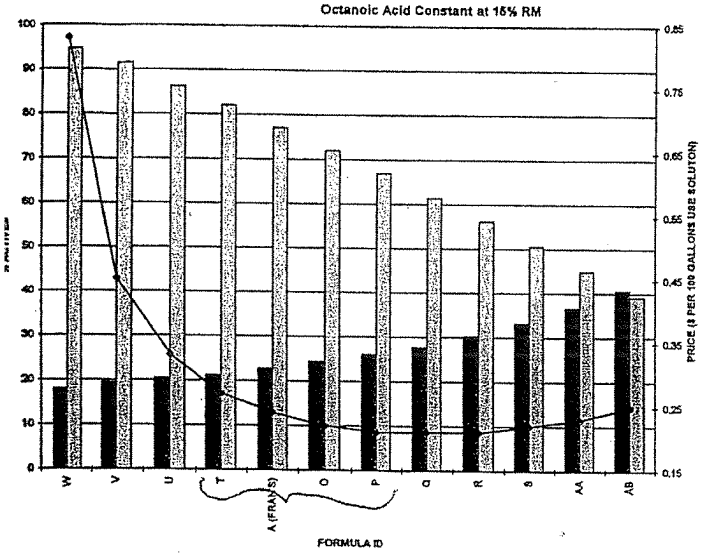
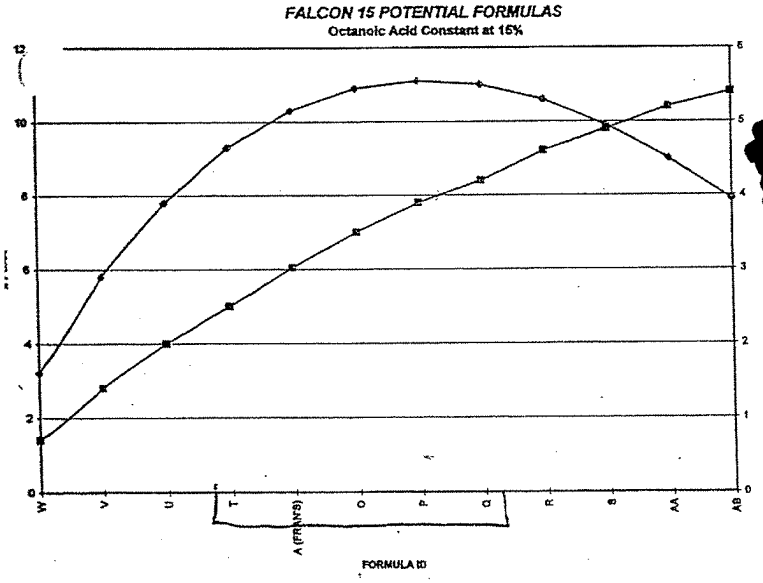
Important Areas of INTEREST WHEN DECIDING UPON A FALCON 15 FORMULA:



	AA	H2O2
W	79%	5%
V	74%	10%
U	69%	15%
T	64%	20%
A(Fran)	59%	25%
O	54%	30%
P	49%	35%
Q	44%	40%
R	39%	45%
S	34%	50%
AA	29%	55%
AB	24%	60%

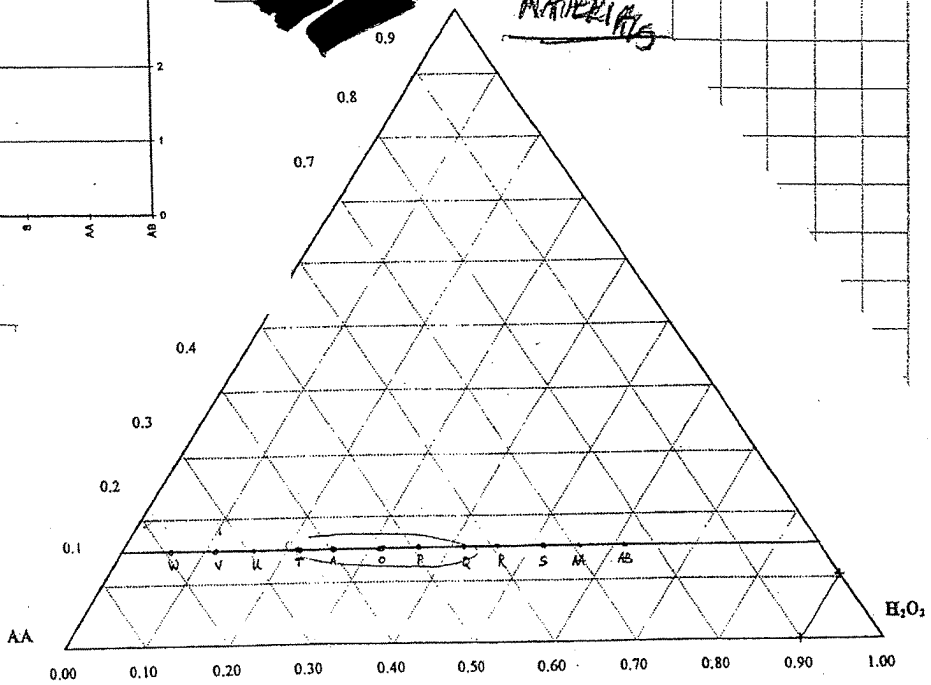
[redacted]

* For Ternary diagram, determine where the detonable formulas are and where the unstable (physically) formulas are.



[redacted]

RAW MATERIALS



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Physical Stability TESTS: MIX ALL SAMPLES WITH A FALCON 15 FORMULA FOR 5 minutes - Stir Plate

CONCENTRATION		TEMP.	QUALITATIVE ANALYSIS
FALCON 15 - CONCENTRATE Prepared by Joel Schilling 9/19/96 SG = 1.0443		100°F	START DATE: 12-27-96
1oz/13 gallons 601 ppm (v/v) 628 (w/w)	Falc = 0.5815 gms H_2O = 925.8 gms	~~Room Temp~~ 60°F	MIX w/ Tap H_2O / A small amount of oil drops seemed to be on the surface, but after mixing Not as evident
1202 ppm (v/v) 1285 ppm (w/w)	Falc = 1.0175 H_2O = 810.2	~~Room Temp~~	Once mixed appears stable
1803 (v/v) 1883 (w/w)	Falc = 1.9075 H_2O = 958.4	60°F ~~Room Temp~~	An obvious oil separation on top before thoroughly mixing. After mixing very slight oil droplets.
2232 (v/v) 2331 (w/w)	Falcon = 1.9020 H_2O = 815.2	60°F ~~Room Temp~~	Oil separation on top before mixing. *obvious oil slick after mixing
6695 (v/v) 6992 (w/w)	Falcon = 5.610 H_2O = 798.2	60°F ~~Room Temp~~	Heavy oil slick prior to mixing and after mixing
9576 1% (w/w)	Falcon = 8.1305 H_2O = 805.1	60°F ~~Room Temp~~	Heavy oil slick before and after mixing.

SEE pp. 40

* Appears that solubility limit for this Falcon 15 ([redacted]) formula is ~~between~~ around 1800 ppm (v/v) at 60°F.

* Could we use a H_2O soluble dye to stand out greater?

$$\frac{10^6}{(X\text{ ppm})(128)} = X \text{ gallons for 1 oz}$$

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Objective: Determine if chemical stability changes substantially over time at elevated temperatures.

SOLUTION: [redacted] Falcon 15 prepared [redacted].

Analytical Results from [redacted]: OA - 8.73%, POOA - 2.67%, H_2O_2 - 4.12%, POAA - 12.30% } LC

FORMULA
59% - AA
25% - H_2O_2 (35%) - 8.75% (100%)
15% - OA
1% - Dequest 2010

Analytical Results

COMPONENTS	[redacted]	Room Temp. Start of Test ([redacted])	Room Temp. End of 1 month (~~Removed from oven~~)	100°F End of 1 month Removed from oven [redacted]
Acetic Acid	N/A	~~59.80~~ 50.67	51.22	63.0%
H_2O_2	4.12%	~~3.80~~ 3.20	1.50	<0.1%
POAA	~~0.36%~~ 11.03%	~~10.20~~ 9.13	7.88	0.40%
POOA	2.67%	~~2.26~~ 2.26	2.24	0.27%
OA	8.73%	~~12.80~~ 12.20	13.0	18.1%
		~~0.6% Dequest~~ 0.6% Dq. ~~11.74% H_2O~~ 21.94% H_2O		

OVEN START DATE: [redacted]
END DATE: [redacted]

Work continued to Page

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Objective: Prepare the following Falcon 15 formulas to determine physical stability of high OA concentrations

FORMULA	Room Temp Stability	40°F Stability (If Room Temp is stable)
X - 59% - AA - 41.04 gms 20% - H_2O_2 - 13.96 20% - OA - 13.98 1% - Dequest C - 0.74	STABLE CLEAR SOLUTION	STABLE CLEAR SOLUTION
Y - 49% - AA - 34.53 gms 30% - H_2O_2 - 21.18 20% - OA - 14.10 1% - Dequest - 0.71	Using 40°F H_2O_2 - when mixing was not stable, but once at room temp. became stable & clear.	NOT STABLE Separated into two layers.
Z - 39% - AA - ~~37.53~~ 26.88 40% - H_2O_2 - ~~27.37~~ 27.37 20% - OA - ~~14.10~~ 0.70 1% - Dequest ~~0.71~~ 13.68	NOT STABLE - Top Octanoic Acid Layer.	Did not test - No need to.

*Depending on stability or instability of these formulas we will vary OA concentration.

Glacial Acetic Acid - 124016 LOT #115371
35% H_2O_2 - 240317 LOT B5762-144
Dequest ADPA - 60AW 250282 LOT# 05036-05 80DR
Octanoic Acid - 190280 LOT 219155

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TITLE PS-15 FALCON 15 **PROJECT NO.** 40

Determine Upper OA Limit. **BOOK NO.** 9980

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According to "Merck Index" Octanoic Acid has a maximum solubility of 0.068 gms / 100 gms H_2O or (680 ppm w/w). It has greater Solubility in acetic acid.

680 ppm (w/w) octanoic acid would be equivalent to

$$\frac{680}{(1.0443)(.1228)} = 5,303 \text{ ppm (v/v) Falcon 15 @ } 25°C \text{ or Room Temperature.}$$

↑ S.G. ↑ % Octanoic Acid by "Solver"

* we noticed solubility problems at 1800 ppm (v/v) although this was at 16°C or 60°F.

* At room temp. we would expect to see slightly better solubility than 5303 due to the coupling ability of acetic acid. Although we are not sure of the solubility limit of peroctanoic acids. - Most likely no limits.

* The samples from pp: 37 were also placed in 40°F refrigerator

All samples use [redacted] Falc. 15 [redacted]	STABILITY 40°F	STABILITY 60°F	STABILITY 70°F
601 ppm (v/v)	STABLE	STABLE - Actual	STABLE
1202	STABLE	STABLE - Raw	STABLE
1803	STABLE	UNSTABLE - Material	STABLE
2232	STABLE	UNSTABLE - Whole	STABLE
6695	UNSTABLE	UNSTABLE - Samples	UNSTABLE
9576	UNSTABLE	UNSTABLE	UNSTABLE

(40°F column, bracketed: Sample of solution Prepared; 70°F column, bracketed: Sample of solution prepared)

*NOTE: The 40°F stability tests were performed by sampling from a larger sample while mixing. The solution may have already separated. Repeat experiment with large Sample

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Additional Concentrates to narrow in on formulas - Determining Physical Stability.

FORMULA	ROOM TEMP. STABILITY	40°F STABILITY
Q AA - 44% - 56.79 gms H_2O_2 - 40% - 51.64 Dequest - 1% - 1.29 OA - 15% - 19.47	NOT STABLE	NOT STABLE
R AA - 39% H_2O_2 - 45% Dequest - 1% OA - 15%	ASSUME INSTABILITY BASED UPON Q & the fact that AA is a coupler	
Y1 AA - 52% - 54.58 gms H_2O_2 - 30% - 31.56 Dequest - 1% - 1.15 OA - 17% - 17.86	STABLE	~~NOT STABLE~~ STABLE
Y2 AA - 49% H_2O_2 - 33 Dequest - 1% OA - 17%	Assume Instability due to P & Y being Unstable - Did not prepare this sample.	
Z1 AA - 39% H_2O_2 - 43% Dequest - 1% OA - 17%	Assume INSTABILITY due to Q being UNSTABLE	
Z2 AA - 42% H_2O_2 - 40% Dequest - 1% OA - 17%	and the fact that AA is a coupler	

RAW MAT'L PRODUCT LOT #'s

* Glacial Acetic Acid: 124016 Lot #115371 Octanoic Acid: 190280 Lot 219615
35% H_2O_2: 240317 Lot B5762-144
Dequest AODA: 250282 Lot #05086-0580DR

Work continued to Page 45

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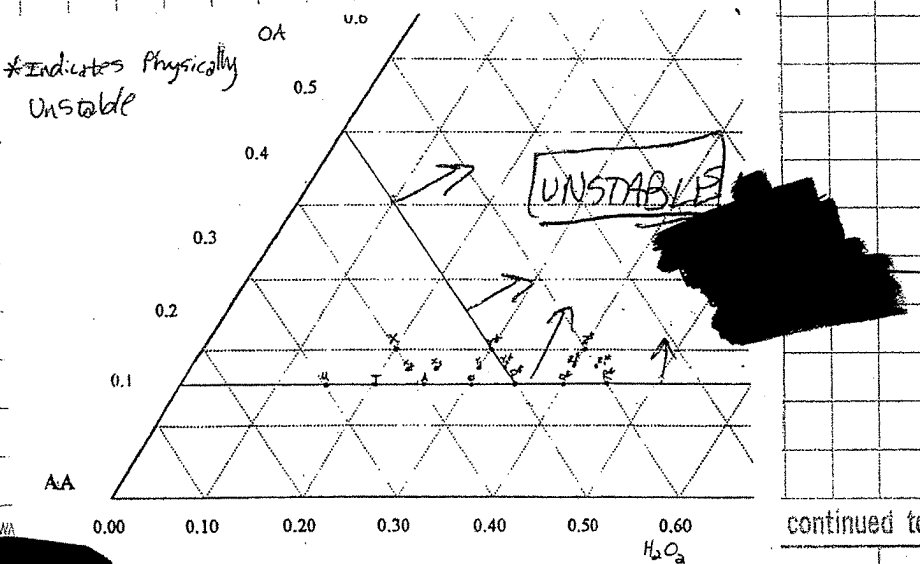
FORMULA	ROOM TEMP STABILITY	40°C STABILITY
P AA - 49% -51.65 H_2O_2 - 35% -37.0 Dequest - 1% -1.05 OA - 15% -15.81	~~UNSTABLE~~ STABLE	UNSTABLE
X_2 AA - 59% - H_2O_2 - 23% - Dequest - 1% - OA - 17% -	Assume STABILITY Based upon Stability of X_1 & X_3.	
X_3 AA - 56% -53.16 H_2O_2 - 26% -24.70 Dequest - 1% -1.00 OA - 17% -16.24	STABLE	STABLE

*STABILITY CURVE THUS FAR

*Indicates Physically Unstable



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*Based upon efficacy data from [redacted] & [redacted] - lower H_2O_2 proves to be more efficacious.

FORMULA	Room Temp. STABILITY	40°F STABILITY
AC: AA - 49.0 - 36.46 gms H_2O_2 - 40.0 - 29.77 Degquest - 1.0 - 0.75 DA - 10.0 - 7.45	STABLE	UNSTABLE
AD: AA - 39.0 - H_2O_2 - 50.0 - Degquest - 1.0 DA - 10.0 -	Did Not Prepare based upon AC - This will be unstable	
AE: AA - 57.0 - 46.04 H_2O_2 - 30.0 - 23.44 Deg. - 1.00 - 0.82 DA - 10.0 - 7.81	STABLE	STABLE
AF: AA - 69.0 - 48.51 H_2O_2 - 20.0 - 14.03 Deg. - 1.0 - 0.74 DA - 10.0 - 7.04	STABLE	STABLE
A: AA - 59.0 - 39.21 H_2O_2 - 25.0 - 16.63 Deg. - 1.0 - 0.66 DA - 15.0 - 10.00	STABLE	STABLE
O: AA - 54.0 - 32.57 H_2O_2 - 30.0 - 18.11 Deg. - 1.0 - 0.62 DA - 15.0 - 9.05	STABLE	STABLE
T: AA - 64.0 - 43.31 H_2O_2 - 20.0 - 13.54 Deg. - 1.0 - 0.69 DA - 15.0 - 10.16	STABLE	STABLE

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FORMULAS SENT TO ANALYTICAL FOR ANALYSIS:

FORMULA ID	DATE Prepared	[redacted]	ANALYTICAL RESULTS		DATE:
		OA ~~Acid~~	Acetic Acid	H_2O_2	POAA	POOA
"AF" AA - 69%, H_2O_2 - 20%, OA - 10%	[redacted]	8.58	58.48	1.79	10.92	1.22
"T" AA - 64%, H_2O_2 - 20%, OA - 15%	[redacted]	11.2	53.28	1.89	10.92	1.65
"X" AA - 59%, H_2O_2 - 20%, OA - 20%	[redacted]	15.8	47.49	1.82	10.11	2.72
"A" AA - 59%, H_2O_2 - 25%, OA - 15%	[redacted]	12.2	48.14	2.98	10.16	2.19
"AE" AA - 59%, H_2O_2 - 30%, OA - 10%	[redacted]	7.45	46.64	3.98	13.46	1.76
"O" AA - 54%, H_2O_2 - 30%, OA - 15%	[redacted]	11.4	40.99	4.15	13.11	2.51

NOTE: AF, T, A, AE & O - Had Equilibration @ room temp for 13 day
X - " " " " " " 27 day

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